CAAP Quarterly Report

Date of Report: October 10, 2016

Contract Number: DTPH56-14-S-N000006

Prepared for: Government Agency: DOT

Project Title: Laser peening for preventing pipe corrosion and failure

Prepared by: Board of Regents of the University of Nebraska for the University of Nebraska-Lincoln

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For quarterly period ending: September 31, 2016

Business and Activity Section

(a) Status Update of Past Quarter Activities

1. Major goal of the project

The ultimate goal of this project is to investigate and develop laser peening of stainless and carbon steels used for pipeline construction to improve their corrosion resistance. The corrosion resistance of pipelines will be enhanced via the compressive residual stress created by laser-induced shock waves during laser peening. It is anticipated that using laser shock peening in the construction of a pipeline will highly improve the reliability, safety, and lifespan of the nation's pipeline transportation system. As stated in our proposal, the major goals of this project will be achieved by organizing the project into four phases, each with specific objectives. Table 1 summarizes the current progress of each phase and its objectives during this reporting period.

Table 1. The completion rate for the objectives of each phase of the project.

Phases	Major Goals and Milestones	Starting Date (mm/dd/yy)	Ending Date (mm/dd/yy)	Completion Rate (%)
Phase 1	1) Develop experimental setup for laser cleaning.	10/01/2014	12/31/2014	100
	2) Establish cleaning mechanism and parameter windows.	01/01/2015	09/30/2016	100
	3) Develop real-time monitoring of cleaning process.	01/01/2015	09/30/2016	no need
Phase 2	1) Develop laser peening system.	10/01/2014	12/31/2014	100
	2) Establish parameter windows for laser peening.	01/01/2015	09/30/2016	100
Phase 3	1) Study surface morphology of the laser-peened surfaces.	01/01/2015	09/30/2016	100
	2) Determine residual stress of pipeline steels after laser cleaning and peening.	01/01/2015	09/30/2016	100
	3) Study corrosion resistance of laser-peened surface.	01/01/2015	09/30/2016	80
Phase 4	Design a laser peening prototype system.	06/30/2016	09/30/2016	100

2. *Specific objectives during this reporting period*

During this reporting period (July 1, 2016, to September 30, 2016), laser cleaning was carried out with a continuous wave (CW) fiber laser. In situ metallographic imaging was performed on carbon steel. In addition, a stress corrosion cracking (SCC) tester designed by our lab was constructed and modified; and portable laser peening equipment was also proposed. The objectives during this reporting period were as follows:

(1) Conduct laser cleaning experiments with a CW fiber laser.

(2) Perform an in situ metallographic investigation of the carbon steel before and after laser peening.

(3) Construct/modify an SCC tester for carbon steel.

(4) Propose portable laser peening equipment for a pipeline system.

3. Significant results

3.1. Experimental setup and procedure

3.1.1. Material preparation

During the reporting period, the samples tested were prepared from 1018 carbon steel (CS) and aluminum-magnesium (Al-Mg) alloy plates, which were cut into a rectangular shape with dimensions of 2 × 2 × 0.25 in (length × width × thickness). Specifically, the Al-Mg alloy plates were used for laser cleaning experiments; and the 1018 CS plates were used for laser peening and metallographic imaging. Prior to the laser peening treatments, the carbon steel surface was mechanically polished using sandpapers of different grades (400-1200 grit) to obtain mirror-like sample surfaces. The polishing was followed by ultrasonic cleaning, first with acetone and then deionized water, to degrease the sample surface. All samples were prepared shortly before the laser shock peening experiments.

3.1.2. Laser cleaning setup and procedures with CW fiber laser

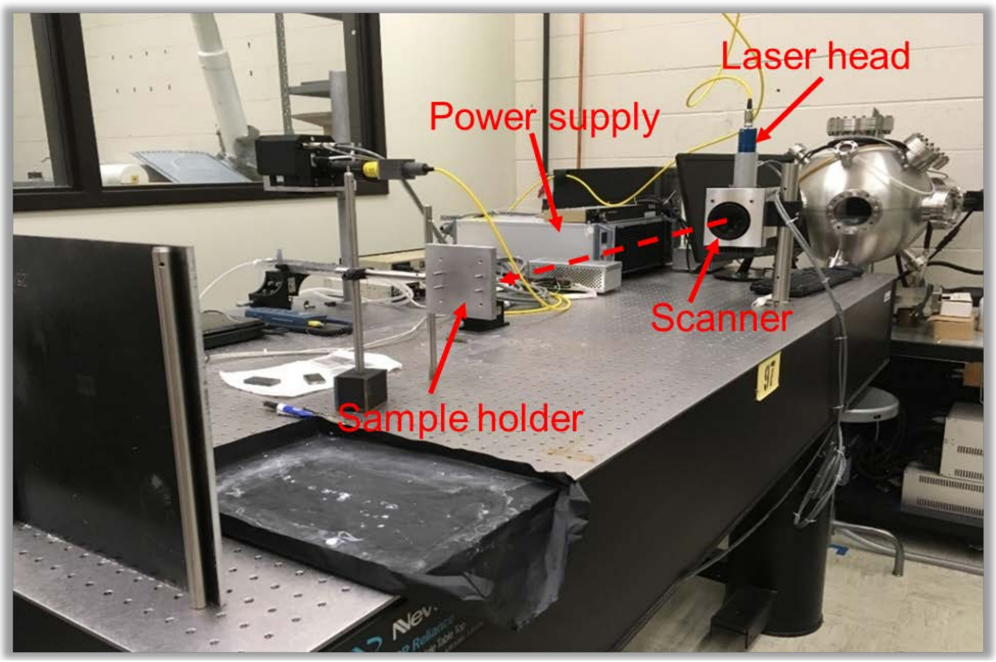

Fig. 1. The laser cleaning system established using a CW fiber laser.

A laser cleaning system was set up using a CW fiber laser (400 W, 1064 nm, IPG Inc.), as shown in Fig. 1. It was more compact compared to the high-power nanosecond fiber laser we used during a previous reporting period (Jan. 2016) and thus preferred for future equipment design. The laser beam was scanned on the contaminated sample surface at a high speed using a galvano scanner for the laser cleaning experiments, in which black paint was used as the contaminative layer. The sample selected for the fiber laser cleaning experiment was an Al-Mg alloy plate, not a carbon steel (CS) plate. Since the Al alloy is more easily damaged by laser cleaning, carbon steel can be better protected if the Al alloy is not ablated.

3.1.3. Low-energy Nd:YAG laser peening setup and procedures

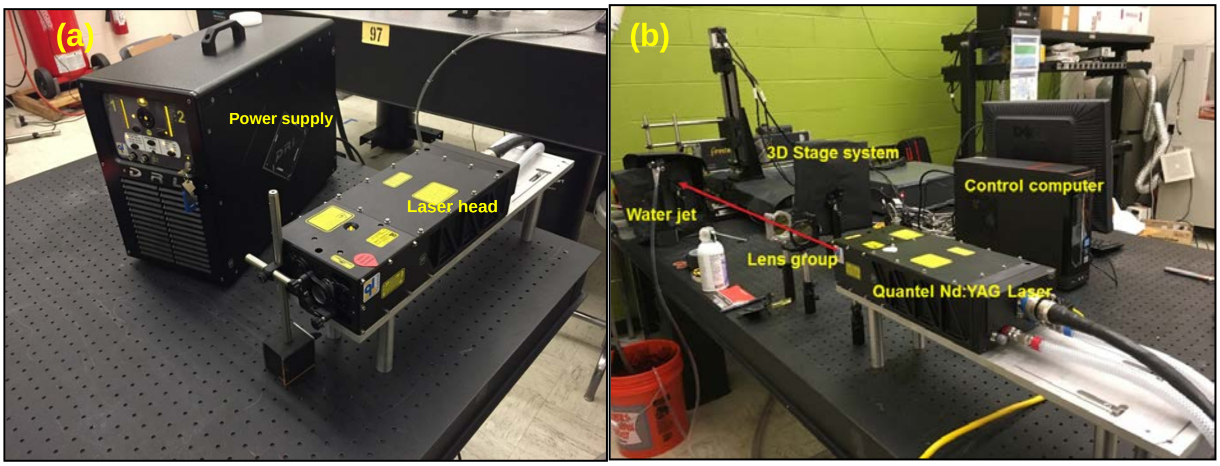

Fig. 2. Low energy Nd:YAG laser peening system.

A laser peening system was established using a low energy compact Quantel Nd:YAG laser (DRL650) and a programmable 3D stage, which will be a test system for the portable laser peening equipment in the future. As shown in Fig. 2a, the weight of the laser is light, with a 6.4 Kg laser head and an 18 Kg power supply. The size of the laser system is also very compact: the laser head measures only 94 × 432 × 160 mm, while the power supply is 406 × 236 × 500 mm. The laser head is completely sealed and filled with high pressure gas allowing it to operate in harsh environments, such as underwater, high humidity, and serious vibration. The main parameters of the laser are: 1064 nm wavelength, 650 mJ maximum pulse energy, 15 ns pulse duration, 6.5 mm output beam diameter, and 1-30 Hz repetition rate. Although the laser repetition rate could be set from 1 to 30 Hz, we only used 5 Hz to avoid the incident laser from being seriously disturbed by the water spray induced by previous laser impacts. The entire laser peening system is shown in Fig. 2b. The sample was held by a 3D stage controlled by a computer to realize the scanning path and speed needed. The water jet was fixed to the stage to keep the water film stable on the sample surface while the stage was moving. A blower was used to prevent water sprays from reaching the optics. The details of the laser parameters/confining layer/sacrificial coating are shown in Table 2.

Table 2. The processing parameters used in LSP experiment.

Laser system	Quantel DRL650 laser; Wavelength: 1064 nm; Pulse energy: 650 mJ; Pulse duration: 15 ns; Frequency: 1-30 Hz; Spot size after focusing: 2.2 or 1.4 mm
Confining layer	Water jet with a layer thickness of approximately 1 mm
Sacrificial coating	Black tape (~100 μm)

3.1.4. Metallographic investigation

To obtain metallographic imaging of the carbon steel, mechanically polished surfaces were further polished by electrochemical polishing, in which the carbon steel was immersed into a specific polishing solution (ethanol solution with 10% $HClO_4$) and positively biased at 25 V for a certain time. After the electrochemical polishing, nital, consisting of 4% HNO_3 and 96% ethanol, was used as the etchant to show the grain structures using a Keyence laser scanning microscope VK-X200K for the image capture.

3.2. Results and discussions

3.2.1. Laser cleaning experiments using a CW fiber laser

A 400 W CW fiber laser with a galvano scanner was employed for laser cleaning of black paint (contamination layer). Since this kind of CW fiber laser does not need a water cooling system, it is smaller in size and weight, which is preferred for the future design of the combined laser peening equipment for pipeline usage.

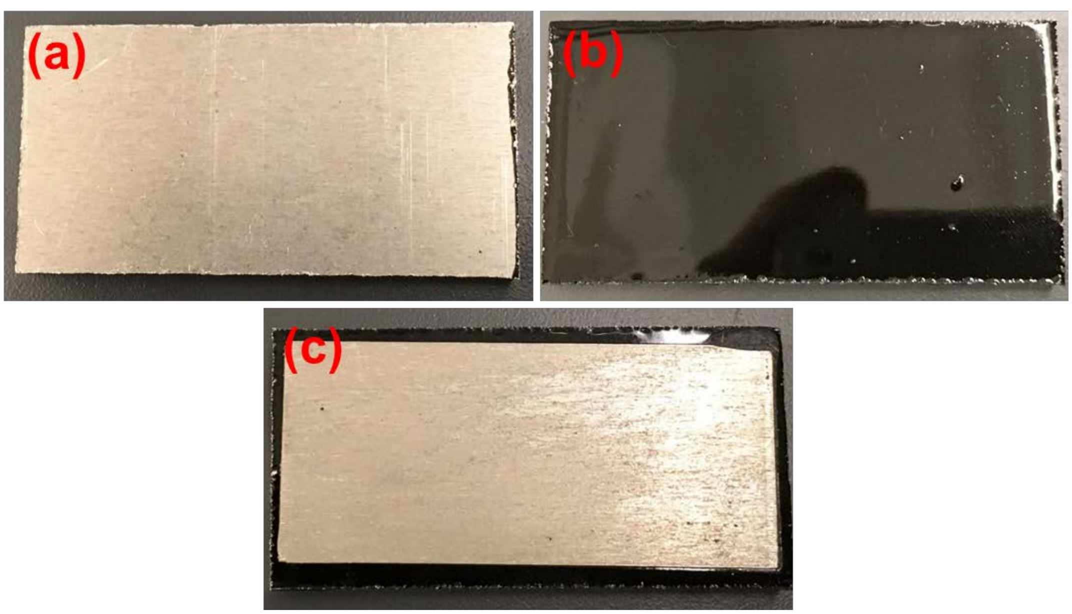

Fig. 3. Optical images of the Al alloy surfaces (a) as-received, (b) covered with black paint, and (c) after laser cleaning using CW fiber laser scanning to remove the black paint.

As shown in Fig. 3., the as-received Al alloy surface (Fig. 3a) was coated with black paint as the contamination layer (Fig. 3b), which was then cleaned with the CW fiber laser with full output power of 400 W. A galvano scanner was employed to scan the laser beam on the sample surface. A scanning speed of 5000 mm/s was used with a focused spot size of 0.5 mm on the sample surface and a working distance of 1.2 m. After a single laser scan, the black paint was removed completely without damaging the Al alloy surface, as shown in Fig. 3c. Since Al alloy is more easily damaged by a laser, the laser power will also work for stainless steel and carbon steel without surface damage. It was demonstrated that the compact 400 W CW fiber laser was able to remove similar surface contamination as did the high-power nanosecond fiber laser, as stated in a previous report (Jan. 2016), and will be used in future equipment designed for pipeline usage.

3.2.2. In situ metallographic investigation of 1018 carbon steel

The microstructural change on the 1018 carbon steel surface, due to the laser shock peening with 100 µm black tape as the sacrificial coating, was further investigated by in situ metallographic imaging (Sec. 3.1.4). Fig. 4 shows three large area metallographic images captured in a similar region of one 1018 carbon steel plate: (a) after polishing and etching; (b) after polishing and etching and laser peening; and (c) after polishing and etching, laser peening, and repolishing and reetching. The grain structure was obvious after polishing and etching, but it was obscured after laser peening. Therefore, electrochemical polishing and etching was performed again to clearly show the microstructural change after laser peening. Since the time spent on electrochemical polishing and etching was very short, the grain structure remained the same for Fig. 4b and 4c. When comparing the large area imaging in Fig. 4a and 4c, slight grain refinement could be observed after laser peening.

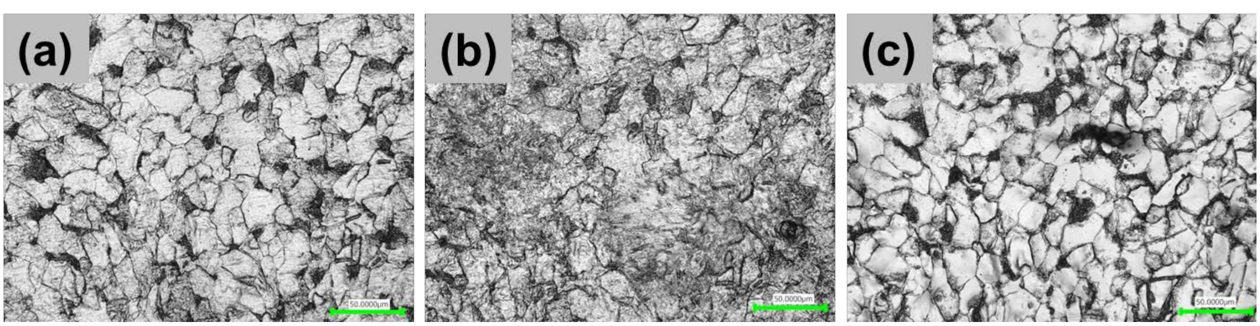

Fig. 4. Large area grain structure on the 1018 carbon steel surface (a) before and (b)(c) after laser peening with black tape (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.6 spot to spot distance, 10 times). (c) After repolishing and reetching

To clarify the grain structure change, in situ metallographic imaging was performed on localized surface regions. In Fig. 5., specific grain structures are outlined in red (a) before and (b) after laser peening. Comparing these structures, we found extra grain boundaries inside the original grains, splitting the large grains into smaller ones, which explains the generation of the smaller grain and grain refinement phenomenon seen in Fig. 4.

In summary, both the large area and localized in situ metallographic imaging revealed grain refinement after laser peening, which could further increase the resistance of the carbon steel to stress corrosion cracking due to the more complicated grain boundaries.

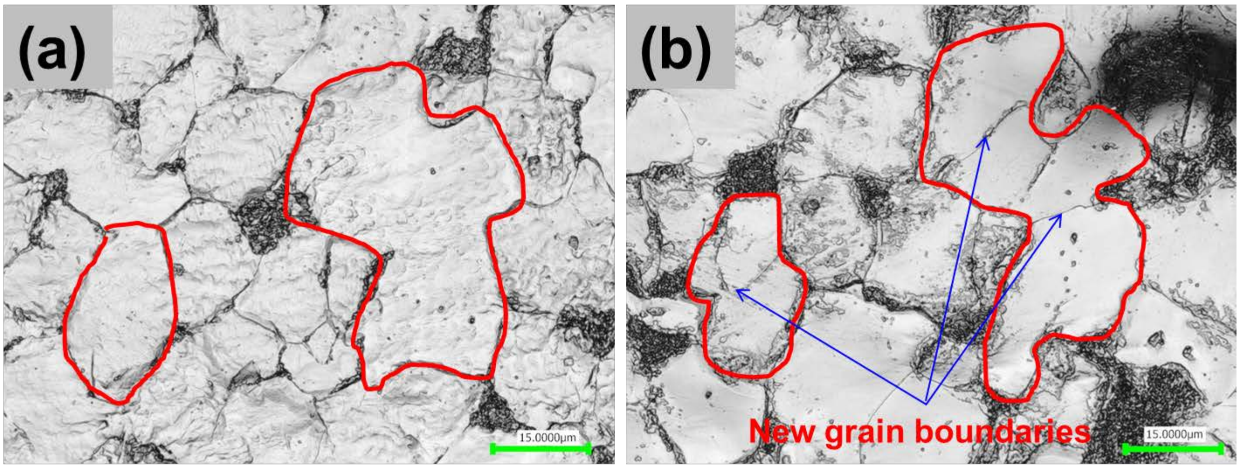

Fig. 5. In-situ grain structure change on the 1018 carbon steel surface (a) before and (b) after laser peening with black tape (1.4 mm spot diameter, 650 mJ pulse energy, 0.5/0.6 spot to spot distance, 10 times). (b) Captured after repolishing and reetching

3.2.3. Lab-designed SCC equipment for carbon steel

It is very hard to determine the SCC resistance improvement of laser-peened carbon steel samples since they easily rust. Thus, a specific SCC tester was designed in our lab. The main parts have been manufactured and installed, as shown in Fig. 6. By tightening the screw at one side of the tester, a constant tensile stress could be applied to the test samples. The samples were machined into a dog-bone shape to fit into the tongs and achieve a uniform stress distribution in the testing area. A dynamometer was used to determine the tensile stress applied to the samples. Two samples were fixed into the tongs: one sample was immersed in a specific SCC test solution, while the other was exposed in air. The solution was heated by a hot plate to provide elevated test temperatures.

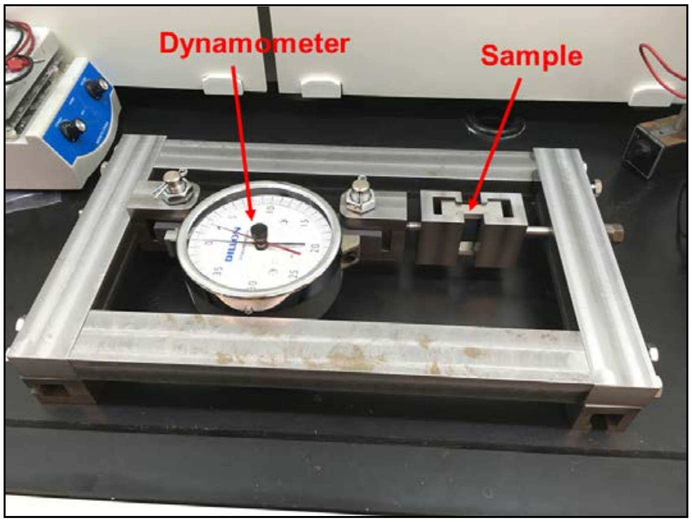

Fig. 6. Optical photo of the preliminary SCC tester (mechanical parts) designed.

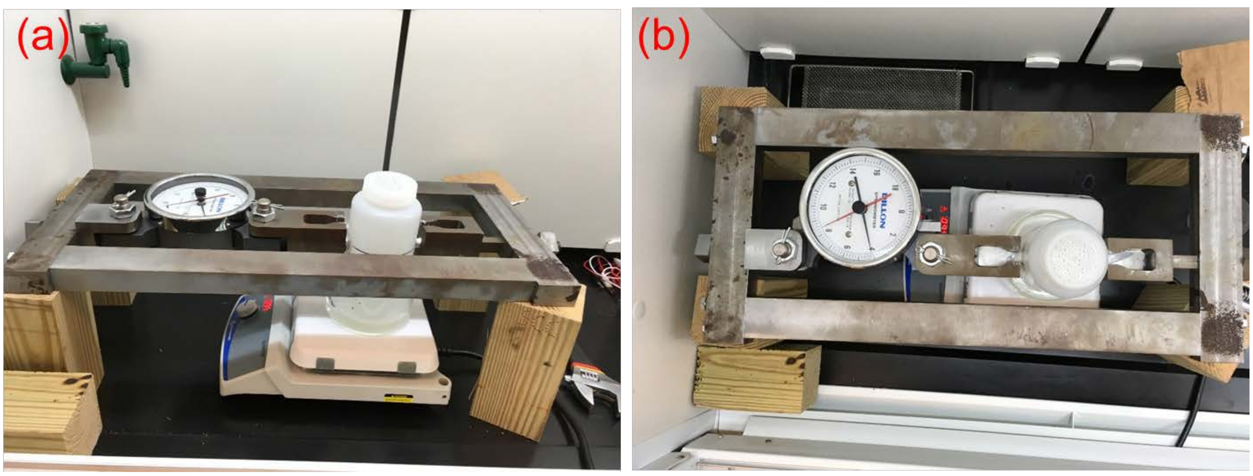

Fig. 7. Optical photo of the improved SCC tester: (a) side view and (b) top view.

After several preliminary SCC tests, some issues were discovered and are summarized as follows: (1) corrosion of the tongs, which also limited the maximum stress applied; (2) insufficient stability of the corrosion temperature; (3) uneven tensile stress distribution due to short sample length; and (4) deformation of the corroded sample leading to a stress concentration of the in-air sample. Therefore, the SCC tester designed was further modified to solve these problems. As shown in Fig. 7, the length of the tester and the sample were extended to obtain a stronger and more uniform pulling force in the test area. The modified clamps could hold one single test sample, which was installed in a plastic bottle to prevent corrosion of the clamps. In addition, the test solution was sealed in the bottle to guarantee the stability of the solution concentration. The solution temperature could also be controlled better by the "water bath" (the water container with hot plate) under the bottle. In summary, the improved SCC tester could apply a tensile stress of up to 500 MPa to the test sample and provide a more stable solution concentration and temperature.

According to several subsequent tests, almost all of the SCC environments could be achieved except the temperature. The maximum heat temperature of the water bath was 100 ^{o}C; but the heat transfer efficiency of the plastic bottle was not enough, resulting in a low environment temperature. Therefore, further modification was carried out. As shown in Fig. 8, the container was changed from a plastic bottle to a stainless steel tank to obtain enough heat transfer efficiency. An oil bath was adopted to improve the heating speed and increase the temperature range of SCC. Professional sealing glue was also employed to guarantee an airtight system even with large sample deformation.

An as-received carbon steel sample is now under testing using the improved SCC tester to determine the best test solution and environment.

Fig. 8. Optical photo of the improved SCC tester with the stainless steel container and oil bath.

3.2.4.   Design proposal for portable laser peening equipment for pipeline systems

The previously reported research results of this project have successfully demonstrated that laser shock peening technology can improve the corrosion resistance of pipeline steels, significantly improving safety, reducing environmental impacts, and enhancing the reliability of the nation's pipeline transportation system. Therefore, the preliminary design of the in-lab laser system is feasible; and it could be fabricated, as shown in Fig. 9. As the in-lab laser peening system uses a high pulse energy (over 2 J) Nd:YAG laser, the whole laser peening system is relatively expensive, bulky (system over 200 kg), and sensitive to the operating environment. Therefore, this system can only be installed in a fixed base, such as workshop or laboratory; and the workpieces need to be transported to the workshop for laser peening before being shipped to the assembly sites.

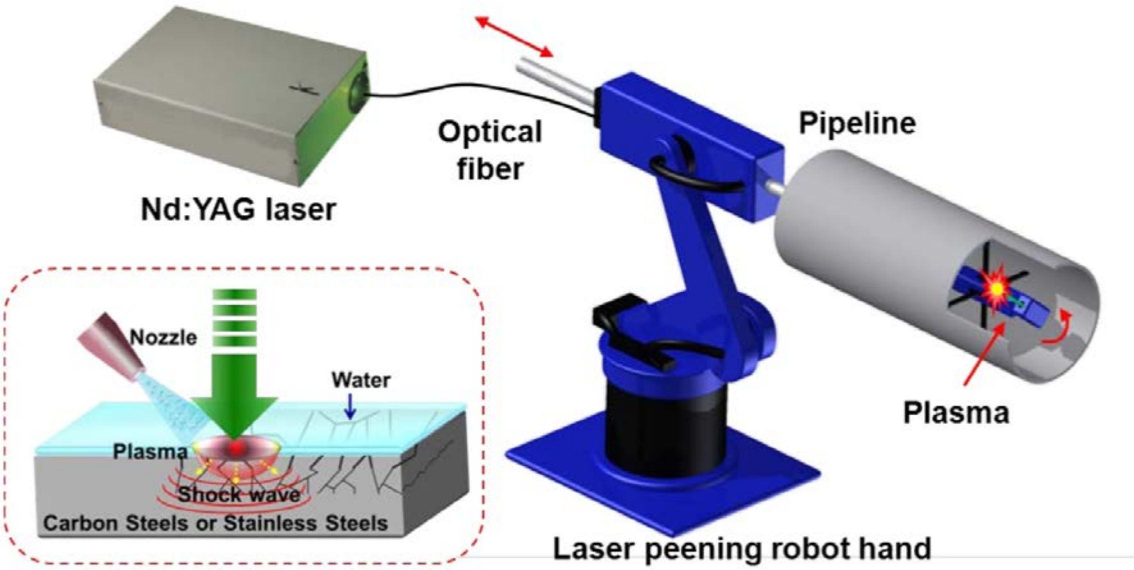

Fig. 9. Preliminary design of the in-lab laser peening equipment for pipeline systems.

However, if compact laser sources with low pulse energy could be used for peening and comparable peening effects could be maintained, all of these shortcomings in laser peening could be overcome.

The PI's group has made a long-term commitment to developing an industrial low energy laser peening technology for Al-Mg alloy. With support from the Office of Naval Research (ONR), we achieved a compressive stress layer of over 200 Mpa (500~2000 μm depth) on the surface of Al-Mg alloy ship plate using a compact Nd:YAG laser with only 0.65 J pulse energy (Fig. 10 a and b). Using a peening process that can be conducted on site, we demonstrated remarkable enhancement of SCC resistance of ship plate. A portable laser peening system for on-site peening of ship plate was also designed.

The PI's group also successfully verified the feasibility of using a low pulse energy laser to peen pipeline steels. By adopting some low energy laser peening technologies developed for Al-Mg ship plate, we observed comparable peening performance on pipeline steels by using a laser pulse energy of only 0.85 J or 0.65 J, as well as SCC resistance improvement, as shown in Fig. 10 c and d.

Thus, further study of the low energy laser peening technologies for pipeline steel and development of on-site high speed laser peening equipment for the pipeline industry may yield better solutions as compared to the in-lab laser peening system developed in this project. Therefore, the PI's group has submitted a new proposal to the DOT for funding the systematic investigation of low energy laser peening technologies to ultimately make low energy laser peening technology industrially reliable for metallic pipelines. The ultimate goal is development of an on-site high speed laser peening prototype system for prevention of pipeline corrosion.

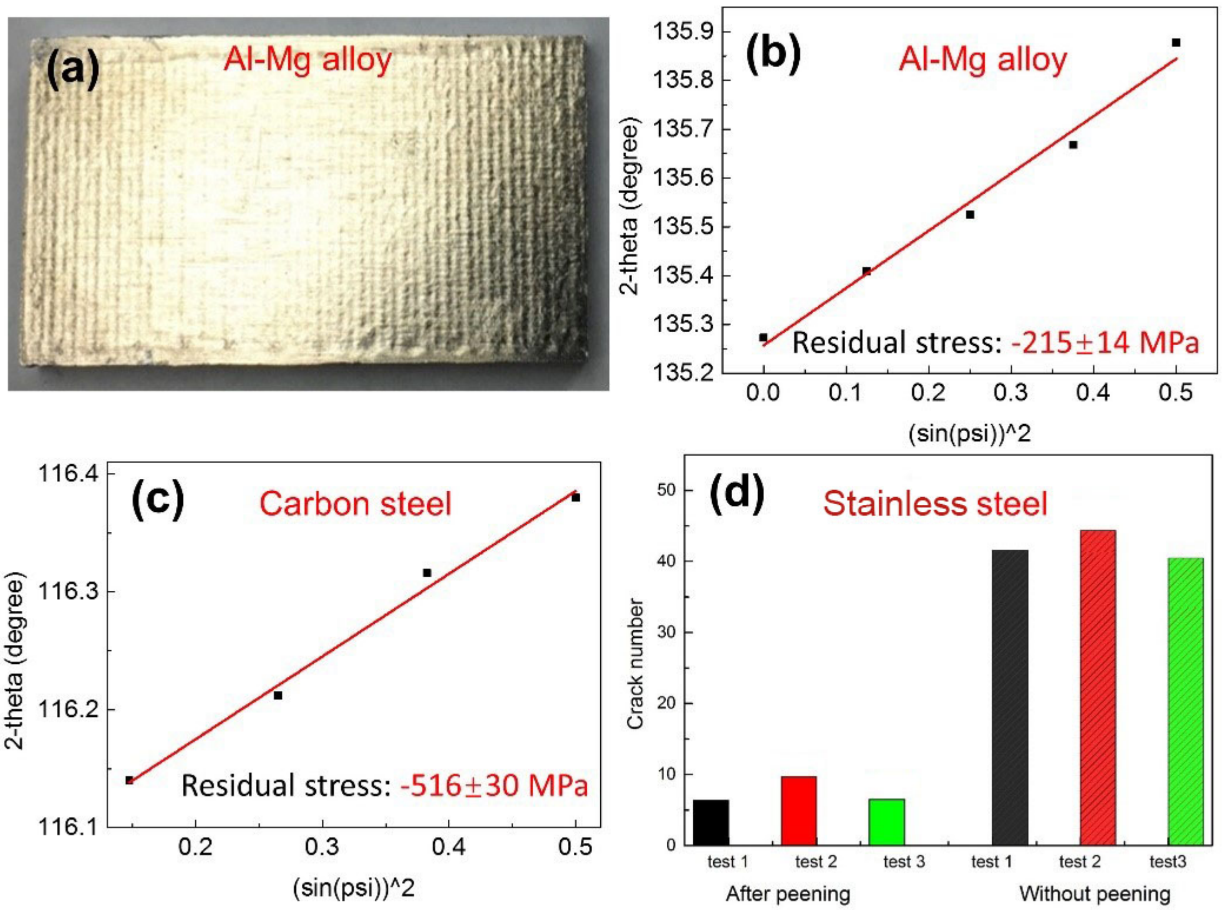

Fig. 10. Low energy laser peening research conducted by the PI's group. (a) and (b) Surface topography and residual stress of Al-Mg alloy surface after peening with 0.65 J laser pulse energy. (c) Residual stress and SCC test results of pipeline steels after peening with 0.85/0.65 J laser pulse energy.

4. Key outcomes

During this reporting period, a CW fiber laser was used for laser cleaning. In situ metallographic investigations were carried out before and after laser shock peening on the surface of 1018 carbon steel. We developed and improved an SCC tester. The design for laser peening equipment for pipeline usage was proposed. Some important conclusions are summarized as follows:

(1) Using a CW fiber laser, a black paint contamination layer was removed without damaging the sample surface, which is preferable for the future design of the combined laser peening equipment.

(2) In situ metallographic imaging of a large area showed grain refinement in 1018 carbon steel before and after laser peening. Localized in situ investigation revealed large grain splitting and smaller grain generation.

(3) The SCC tester designed in our lab has been manufactured and improved to provide stronger/uniform stress, solution concentration, and environmental temperature.

(4) The original design of the laser peening equipment is applicable for in-factory operation, according to the results of our previous experiments. A portable laser peening system is proposed for in-field processing. The creation of specific equipment to improve corrosion resistance of pipeline materials based on our design for the Navy is promising with further financial support.

(b) Description of any problems/challenges

No problems were experienced during this reporting period.

FEDERAL FINANCIAL REPORT

(Follow form instructions)

1. Federal Agency and Organizational Element to Which Report is Submitted U.S. DEPARTMENT OF TRANSPORTATION	2. Federal Grant or Other Identifying Number Assigned by Federal Agency (To report multiple grants, use FFR Attachment) DTPH5614HCAP07	Page 1 of 1 pages

3. Recipient Organization (Name and complete address including Zip code)

UNIVERSITY OF NEBRASKA-LINCOLN 151 WHITTIER RESEARCH CENTER, 2200 VINE STREET LINCOLN, NE 68583-0861

4a. DUNS Number	4b. EIN	5. Recipient Account Number or Identifying Number (To report multiple grants, use FFR Attachment)	6. Report Type	7. Basis of Accounting
55-5456995	47-0049123	25-1109-0136-001	☐ Quarterly ☐ Semi-Annual ☑ Annual ☐ Final	☑ Cash ☐ Accrual

8. Project/Grant Period (Month, Day, Year)		9. Reporting Period End Date (Month, Day, Year)
From: 9/30/2014	To: 9/29/2017	9/30/2016

10. **Transactions**	Cumulative
(Use lines a-c for single or combined multiple grant reporting)	
Federal Cash (To report multiple grants separately, also use FFR Attachment):	
a. Cash Receipts	31,589.10
b. Cash Disbursements	35,345.47
c. Cash on Hand (line a minus b)	(3,756.37)
(Use lines d-o for single grant reporting)	
Federal Expenditures and Unobligated Balance:	
d. Total Federal funds authorized	100,000.00
e. Federal share of expenditures	35,345.47
f. Federal share of unliquidated obligations	-
g. Total Federal share (sum of lines e and f)	35,345.47
h. Unobligated balance of Federal funds (line d minus g)	64,654.53
Recipient Share:	
i. Total recipient share required	66,352.00
j. Recipient share of expenditures	29,818.32
k. Remaining recipient share to be provided (line i minus j)	36,533.68
Program Income:	
l. Total Federal share of program income earned	
m. Program income expended in accordance with the deduction alternative	
n. Program income expended in accordance with the addition alternative	
o. Unexpended program income (line l minus line m or line n)	

11. Indirect Expense	a. Type	b. Rate	c. Period From	Period To	d. Base	e. Amount Charged	f. Federal Share
	Predetermined	51%	7/1/2013	6/30/2015	22,618.71	11,535.54	11,535.54
				g. Totals:	22,618.71	11,535.54	11,535.54

12. Remarks: Attach any explanations deemed necessary or information required by Federal sponsoring agency in compliance with governing legislation:

13. Certification: By signing this report, I certify to the best of my knowledge and belief that the report is true, complete, and accurate, and the expenditures, disbursements and cash receipts are for the purposes and intent set forth in the award documents. I am aware that any false, fictitious, or fraudulent information may subject me to criminal, civil, or administrative penalties. (U.S. Code, Title 18, Section 1001)

a. Typed or Printed Name and Title of Authorized Certifying Official Deb Arent, Assistant Director	c. Telephone (Area code, number, and extension) 402-472-6327
	d. Email Address darent1@unl.edu
b. Signature of Authorized Certifying Official Deb Arent Digitally signed by Deb Arent DN: cn=Deb Arent, o=University of Nebraska-Lincoln, ou=Office of Sponsored Programs, email=darent1@unl.edu, c=US Date: 2016.10.11 05:49:11 -05'00'	e. Date Report Submitted (Month, Day, Year) 10/3/2016
	14. Agency use only:

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OMB Approval Number: 0348-0061
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